

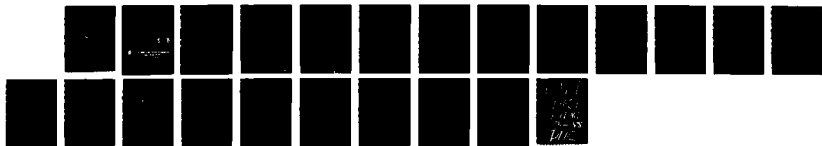
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TECHNICAL REPORT ARAED-TR-87031

**1, 2, 3-TRIAMINOGUANIDINIUM NITRATE: AN ALTERNATE
EXPLANATION OF THE NITRATE ANION DISORDER**

A. J. BRACUTI

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<p>The molecular structure of triaminoguanidinium nitrate was redetermined on the basis of a positionally disordered nitrate ion model and compared with results from the model that assumes dynamic rigid-body motion in the nitrate ion. It was concluded that at room temperature the nitrate ion is undergoing rigid-body motion. It was also recommended that a low temperature structure determination be carried out in conjunction with thermal studies in order to fully characterize nitrate ion motion-disorder.</p> <p>(Key words:)</p>					
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INTRODUCTION

Triaminoguanidiinium nitrate (TAGN) has the property of accelerating propellant burning rates when added to cyclic nitramine (RDX) formulations. This is of great importance to the DoD because of current Army efforts to develop high performance LOVA propellants containing RDX. Since this property may be directly related to its molecular structure, the structure determination of TAGN is a necessary step in understanding its function as a burning rate accelerator.

In previous TAGN structure determinations (refs 1 and 2), extraordinarily large anisotropic thermal vibration were observed for the oxygen atoms of the nitrate ion. Since large thermal vibrations can be the harbingers of dynamic disorder which sometimes may be caused by rigid-body motion, it was suggested that the nitrate ion behaved as a rigid-rotor hindered from free rotation by the interactive forces of hydrogen bonding.

Instead of assuming dynamic or continuous rigid-body rotation, this disorder can also be explained on the basis of random orientation about a rotation axis. Specifically, the nitrate ion contains a three-fold axis that is perpendicular to the plane of the ion and passes through the nitrogen atom. In this model, a static condition exists where the nitrate ion has more than one possible orientation in which oxygen atoms can be placed. Because the number of atomic sites created by this model exceeds the number of oxygen atoms contained in this compound, it must be assumed that throughout the crystal the atom sites are incompletely filled. In this way, the resultant x-ray diffraction pattern is a composite picture of all the possible orientations. This static model is analogous to a motion picture film in which a series of rapidly flashing still-pictures of an object in different orientations create the illusion of motion for that object. Therefore, both the dynamic rigid-rotor and static models would account for the disorder and produce similar x-ray diffraction patterns.

The static disorder model can be treated by assuming that either the atomic sites are randomly filled throughout space or that each site is partially but not necessarily equally filled. From a computational point of view the later assumption is much easier to handle.

It must be emphasized, however, that there is an implicit assumption in the static model that at some higher temperature it is necessary for the oxygen atoms to rotate either freely or in a hindered fashion about the three-fold axis in order to attain a static state of disorder observed at a lower temperature. Therefore, the real question to answer is whether the oxygen atoms continually rotate at room temperature or are they frozen in a disordered state caused by rotation at a higher temperature.

Since the room temperature disorder displayed by the nitrate ion has not been unequivocally resolved, a new structure investigation was initiated in which the main thrust was focused on examining positional disorder. This phase of the investigation will address the static model and compare the results with those previously obtained assuming continual rigid-body motion.

DISCUSSION

Data Collection

A colorless acicular crystal of TAGN having approximate dimensions of 0.10 x 0.15 x 0.30 mm was mounted on a glass fiber with the longest axis approximately parallel to the phi axis of the goniometer. The preliminary examination and data collection were performed with Cu K α x-radiation ($\lambda = 1.54184$ Å) on an Enraf-Nonius DAD4 computer controlled kappa axis monochromator.

Cell constants and orientation matrix for data collection were obtained from least-squares refinement. As a check on crystal, quality omega scans of several intense reflections were measured. The width at reflection intensity half-height was 0.20 deg with a take-off angle 5.8 deg which indicates good crystal quality.

The intensity data were collected at a temperature of 23°C \pm 1°C using the ω - θ technique with a scan rate varying from 2 to 25°C/min (in omega). The variable scan rate allows rapid data collection of intense reflections and also assures good counting statistics for weak reflections where a slow scan should be used. Intensity data for 1599 reflections of which 783 were unique and not systematically absent with $h = 0$ to 10, $k = 0$ to 15, and $l = 0$ to 8 were collected to a maximum 2θ value of 150.0. The scan range (in deg) was determined as a function of θ to correct for the separation of the K α doublet (ref 3). The scan width was calculated by the equation

$$\theta \text{ scan width} = 0.7 + 0.300 \tan \theta$$

All background counts were measured with the moving-crystal technique by scanning an additional 25% above and below this range. Therefore, the ratio of peak counting-time to background counting-time was 2:1. The horizontal aperture width ranged from 2.0 to 5.7 mm while the vertical aperture was set at 2.0 mm. The diameter of the incident beam collimator was set at 0.7 mm and the crystal-to-detector distance was 21 cm. For intense reflections, an attenuator (attenuation factor of 15.5) was automatically inserted in front of the detector.

Data Reduction

Lorentz-polarization and secondary extinction corrections (ref 4), but no absorption corrections, were applied to the intensity data. The final extinction coefficient was refined in least squares to a value of 0.0000124 (in absolute units). The intensities of equivalent reflections were averaged. The agreement factors for the averaging of the 757 observed reflections was 5.7% based on intensity and 4.3% based on F_o .

The crystal data are:

Orthorhombic, space group $Pbcm$ (#57), $a = 8.366(2)$, $b = 12.649(2)$,
 $c = 6.556(1)$ Å, $V = 693.8$ Å³, $Z = 4$, $D_x = 1.60$ g/cm³, $\rho = 12.9$ g/cm³
 $F(000) = 352$, $F_w = 167.13$, $T = 296$ K (23°C), systematic absences: $0k\lambda$, $k = 2n + 1$; $h0\lambda$, $\lambda = 2n + 1$.

On the basis of systematic absences and E probability distribution statistics, the centrosymmetric space group $Pbcm$ was selected.

Structure Determination

The structure was solved by direct methods (Enraf-Nonius Structure Determination Package, ref 3). Using 175 reflections (minimum E of 1.23) and 2529 relationships, a total of 32 phase sets were produced. Ten of the nonhydrogen atoms were located from an E-map prepared from the phase set with the following best probability statistics: absolute figure of merit 1.39, residual 0.26, and psi zero 0.690. The remaining atoms were located in successive difference Fourier syntheses. The hydrogen atoms were refined isotropically while the nonhydrogen atoms were refined anisotropically by full-matrix least-squares in which the function $\sum w(|F_o| - |F_c|)^2$ was minimized and w is defined as $4F_o^2/\sigma^2(F_o^2)$. The standard deviation on intensities, $[\sigma^2(F_o^2)]$, is defined

$$\sigma^2(F_o^2) = [S^2 (C + R^2B) + (\rho F_o^2)^2] / (Lp)^2$$

in which R is the scan rate, C is the total integrated peak count, B is the ratio of the scan time to the background count, Lp is the Lorentz and polarization factor, and the parameter ρ is a factor introduced to downweight the intense reflections. In this case ρ was set to 0.060.

The x-ray scattering factors were taken from Cromer and Waber (ref 5). Anomalous dispersion effects were included in the calculation of structure factors (ref 6), and the values of $\Delta f'$ and $\Delta f''$ were those of Cromer (ref 7). Only 492 reflections having intensities greater than 3.0 times their standard deviations were used in the refinements.

The final cycle of refinement included 98 variables and converged (largest parameter shift was 0.08 times its estimated standard deviation) with an unweighted agreement factor of $R = 0.58$

$$R = \Sigma (|F_o| - |F_c|) / \Sigma |F_o|$$

and weighted agreement factor of $R_w = 0.073$ in which

$$R_w = \text{SQRT} [\Sigma w(|F_o| - |F_c|)^2 / \Sigma w F_o^2]$$

The standard deviation of an observation unit weight was 1.83. The highest peak in the final difference Fourier had a height of 0.25 e/A with an estimated error based on ΔF (ref 8) of 0.07.

Description of the Crystal Structure

The atomic coordinates and thermal parameters of the title compound are presented in tables 1 through 3. The molecular structure of TAGN, as expected, agrees quite closely with the structure reported in earlier investigations (refs 3 and 4). The structure consists of layers of ions oriented parallel to (001) located at $1/4 c$. Within each layer, ionic forces and hydrogen bonds prevail, while the layers are held together by van der Waal's forces and hydrogen bonds. The structure is depicted in figure 1 and is also presented as stereo pairs of unit cells for three-dimensional viewing in figure 2.

Description of the TAG Cation

The bond lengths and bond angles for this cation are listed in tables 4 and 5, respectively. The mean bond lengths for C--N [1.322 (4) Å] and the N--N [1.413 (4) Å] agree with the previously reported values (refs 1 and 2). The molecular conformation of this cation (C_{3h}) is presented in figure 3.

Description of the Nitrate Anion

In this structure, each nitrate ion should be located on a mirror plane with its center of gravity, a nitrogen atom, lying in the mirror plane with a coplanar three-fold rotation axis passing through it. This rotational axis is a symmetry requirement of the nitrate ion

(D_{3h} point group) rather than a symmetry requirement of the space group of the structure. However, the ionic plane is normal to the mirror plane. These molecular and crystal structure symmetry operators impose the following constraints on the positions of the oxygen atoms:

1. Each oxygen atom must generate another every 120 deg and N--O bond lengths should be equivalent (molecular symmetry condition only)
2. Each oxygen atom out of the mirror must generate a mirror twin (space group symmetry condition)
3. All oxygen atoms must be in the same plane

The nitrate anion (fig. 3) along with the atom numbering scheme exhibits oxygen atom disorder which approximates a molecular conformation consistent with these symmetry constraints.

Close inspection of figure 3 reveals that the nitrate anion has two sets of oxygen triads with all oxygen atoms positioned out of the mirror plane. If one oxygen atom were in the mirror, this would have resulted in an odd number of sets of oxygen triads. When performing the three-fold rotational symmetry operation, atom O(1) produces atom O(2B) which generates atom O(2A) which regenerates O(1) to complete the 360 rotation. Each oxygen atom then reflects across the mirror plane to generate its twin. Since there are six oxygen atom positions surrounding one nitrogen atom position, the observed stoichiometry requires that each oxygen position throughout the crystal must be half-filled. This 50% occupational factor also was obtained independently from the x-ray diffraction analysis calculations.

The observed bond lengths for the anion listed in table 4 are 1.283(4), 1.097(9), and 1.333(10) for N(7)---O(1), N(7)---O(2A), and N(7)---O(2B), respectively. Only the N(7)---O(1) bond length is in agreement with the N---O bond lengths previously reported (refs 1 and 2) where continuous rigid-body motion was assumed. Not only do the divergent values for N(7)---O(2A) and N(7)---O(2B) disagree with those previously reported values, their significant inequality also distorts the anticipated three-fold molecular symmetry of the nitrate ion. The bond angles also deviate significantly from 120 as seen in table 5 in which O(2B)---N(7)---O(1), O(2A)---N(7)---O(1), and O(2B)---N(7)---O(2A), are 110.7(5), 127.9(5), and 122.0(5), respectively.

CONCLUSIONS AND RECOMMENDATIONS

It was concluded that at room temperature the nitrate ion is undergoing rigid-body molecular motion. This conclusion was based on the following observations:

- The NO₃ conformation observed in the dynamic model does not violate D_{3h} point group symmetry and agrees with the accepted conformation
- The N---O bond lengths observed in the dynamic model are equivalent and consistent with accepted N---O bond lengths.
- With the static positional disorder model, the ionic conformation is distorted
- The N---O bond lengths in this static model are not equivalent and not consistent with accepted N---O bond distances
- The oxygen atoms still display large atomic thermal vibrations in the static model

It is recommended that a low temperature x-ray diffraction study in conjunction with the DSC investigation be carried out in order to better characterize the nitrate motion and determine the thermal mechanism. In addition, a complete low temperature electron density determination should be undertaken.

Table 1. Atomic coordinates and their estimated standard deviations

<u>Atom</u>	<u>x</u>	<u>y</u>	<u>z</u>
O(1)	0.3601(4)	0.0450(3)	0.3170(7)
O(2A)	0.2045(6)	0.1104(4)	0.4092(7)
O(2B)	0.1580(6)	0.1385(4)	0.4054(9)
N(1)	0.6665(4)	0.1418(3)	0.25
N(2)	0.7793(4)	0.0585(3)	0.25
N(3)	0.8749(4)	0.2592(3)	0.25
N(4)	0.9237(4)	0.3654(3)	0.25
N(5)	0.6172(4)	0.3188(3)	0.25
N(6)	0.4512(4)	0.2957(3)	0.25
N(7)	0.2403(3)	0.0980(3)	0.25
C(1)	0.7185(4)	0.2400(3)	0.25
H(1)	0.564(6)	0.118(4)	0.25
H(2)	0.759(3)	0.018(3)	0.147(5)
H(3)	0.936(5)	0.210(4)	0.25
H(4)	0.985(3)	0.373(2)	0.143(4)
H(5)	0.637(5)	0.391(3)	0.25
H(6)	0.412(3)	0.328(3)	0.361(5)

Table 2. Anisotropic and equivalent temperature factors and their estimated standard deviations for nonhydrogen atoms

<u>Atom</u>	<u>B(11)</u>	<u>B(22)</u>	<u>B(33)</u>	<u>B(12)</u>	<u>B(13)</u>	<u>B(23)</u>	<u>B_{eq}</u>
TAG CATION							
N(1)	2.61(1)	3.5(1)	4.8(2)	0.2(1)	0.0	0.0	3.64(7)
N(2)	3.5(1)	3.7(1)	5.2(2)	0.5(1)	0.0	0.0	4.15(8)
N(3)	2.2(1)	4.6(2)	5.4(2)	-0.1(1)	0.0	0.0	4.08(8)
N(4)	2.9(1)	5.6(2)	5.3(2)	-0.7(1)	0.0	0.0	4.61(9)
N(5)	2.5(1)	3.8(1)	5.1(2)	-0.1(1)	0.0	0.0	3.78(8)
N(6)	2.3(1)	4.6(2)	5.4(2)	0.2(1)	0.0	0.0	4.11(8)
C(1)	2.9(1)	3.8(2)	2.5(1)	0.1(1)	0.0	0.0	3.03(7)
Nitrate anion							
N(7)	2.1(1)	3.2(1)	5.6(2)	-0.2(1)	0.0	0.0	3.64(7)
O(1)	2.6(1)	4.1(2)	7.9(4)	0.3(1)	-0.6(2)	0.5(2)	4.9(1)
O(2A)	5.8(2)	6.1(2)	3.6(2)	-0.6(2)	1.9(2)	-0.9(2)	5.2(1)
O(2B)	4.3(2)	6.8(3)	7.2(3)	0.7(2)	2.3(2)	-1.7(2)	6.1(1)

The form of the anisotropic temperature factor is:

$$\exp[-0.25(h^2 a^2 B(11) + k^2 b^2 B(22) + l^2 c^2 B(33) + 2hkabB(12) + hlacB(13) + klbcB(23))]. \quad B_{eq} = 1/3[B(11) + B(22) + B(33)]$$

Table 3. Isotropic temperature factors and their estimated standard deviations for the hydrogen atoms

<u>Atom</u>	<u>B</u>
H(1)	7.7(15)
H(2)	5.3(8)
H(3)	6.7(4)
H(4)	3.1(6)
H(5)	3.6(9)
H(6)	5.7(8)

Table 4. Bond lengths and their estimated standard deviations

<u>Atom 1</u>	<u>Atom 2</u>	<u>Distance</u>
C(1)	N(1)	1.316(4)
C(1)	N(3)	1.330(4)
C(1)	N(5)	1.309(4)
N(1)	N(2)	1.415(4)
N(1)	H(1)	0.91(5)
N(2)	H(2)	0.87(3)
N(3)	N(4)	1.404(5)
N(3)	H(3)	0.80(5)
N(4)	H(4)	0.87(2)
N(5)	N(6)	1.419(4)
N(5)	H(5)	0.93(4)
N(6)	H(6)	0.90(3)
N(7)	O(1)	1.283(4)
N(7)	O(2A)	1.097(9)
N(7)	O(2B)	1.333(10)

Table 5. Bond angles and their estimated standard deviations

<u>Atom 1</u>	<u>Atom 2</u>	<u>Atom 3</u>	<u>Angle</u>
N(1)	C(1)	N(3)	119.8(3)
N(1)	C(1)	N(5)	120.3(3)
N(3)	C(1)	N(5)	119.9(4)
N(2)	N(1)	C(1)	118.9(3)
N(4)	N(3)	C(1)	117.4(3)
N(6)	N(5)	C(1)	118.5(3)
N(2)	N(1)	H(1)	112.0(3)
N(4)	N(3)	H(3)	124.0(3)
N(6)	N(5)	H(5)	112.0(2)
C(1)	N(1)	H(1)	129.0(3)
C(1)	N(3)	H(3)	119.0(3)
C(1)	N(5)	H(5)	129.0(2)
N(1)	N(2)	H(2)	108.0(2)
N(3)	N(4)	H(4)	106.0(2)
N(5)	N(6)	H(6)	105.0(2)
H(2)	N(2)	H(2)	103.0(4)
H(4)	N(4)	H(4)	107.0(3)
H(6)	N(6)	H(6)	105.0(2)
O(1)	N(7)	O(2A)	127.9(5)
O(1)	N(7)	O(2B)	110.15
O(2A)	N(7)	O(2B)	122.0(5)

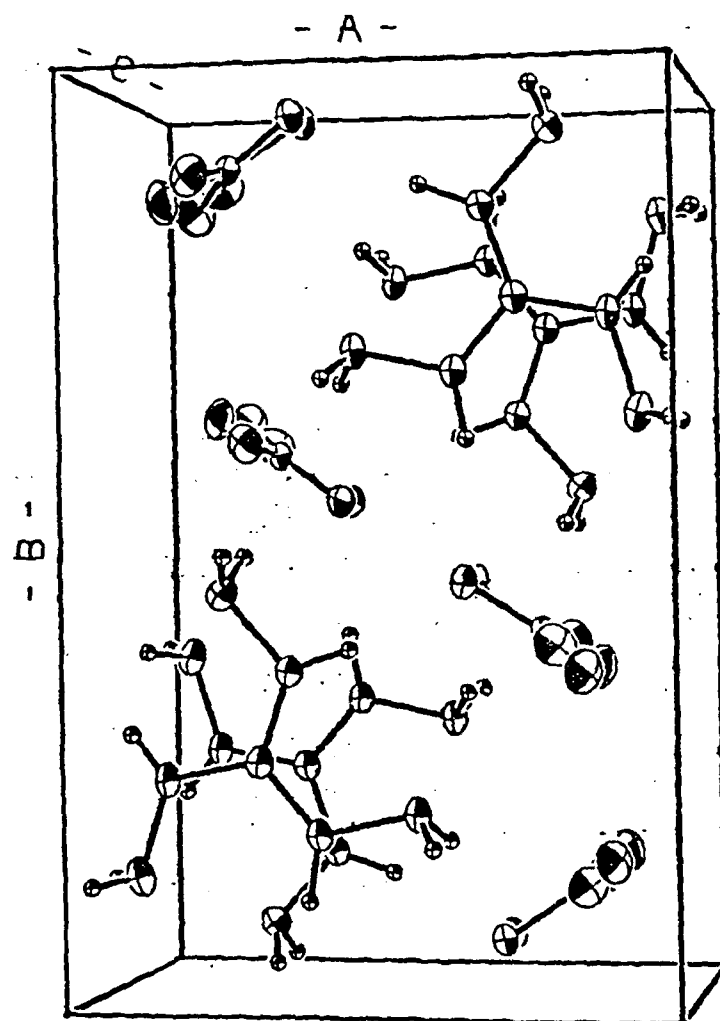


Figure 1. Unit cell of triaminoguanidinium nitrate
viewed down the c axis

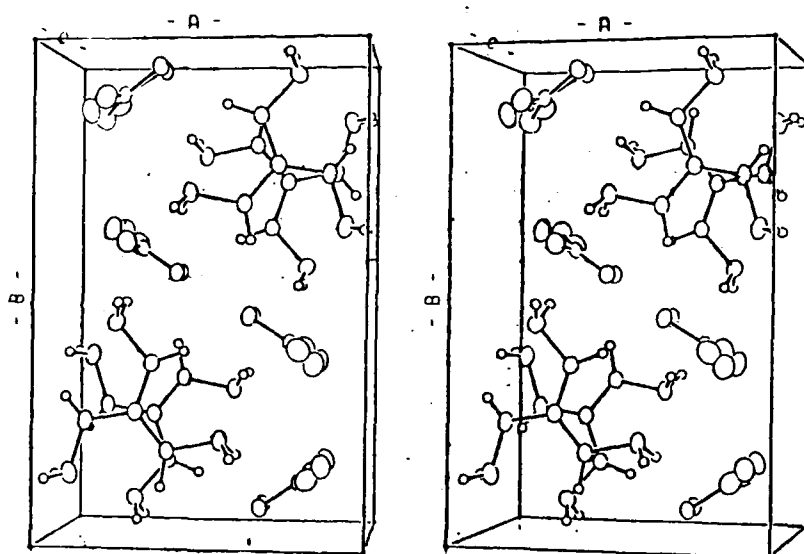


Figure 2. Stereo pair of triaminoguanidinium nitrate unit cells viewed down the c axis

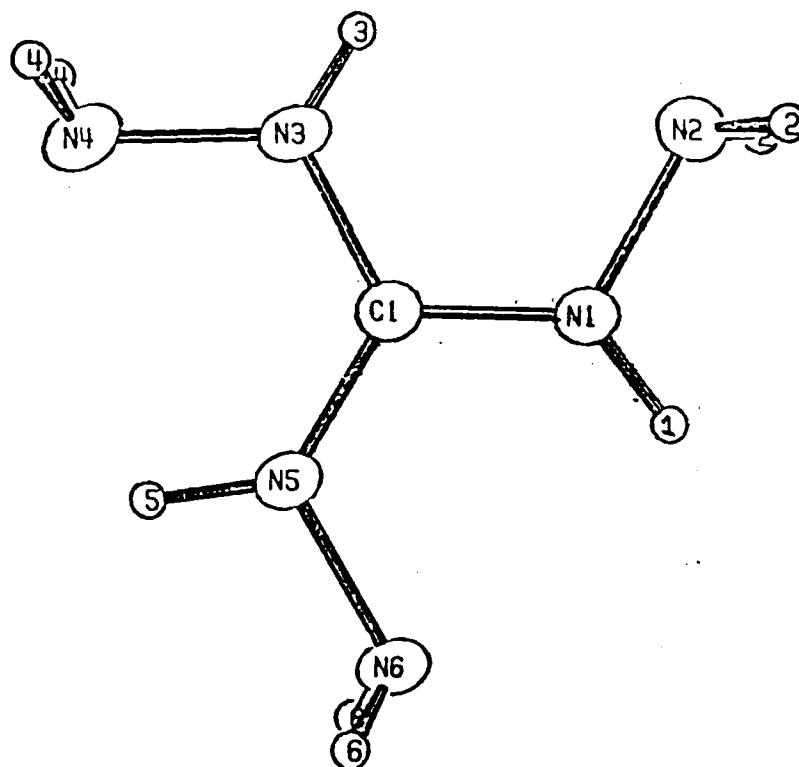


Figure 3. Molecular conformation of triaminoguanidinium cation

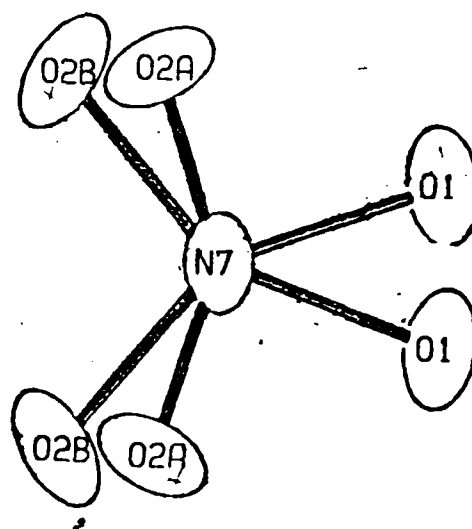


Figure 4. Molecular conformation of disordered nitrate anion

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